CONTRACT REPORT NO. OST 84-00166

- RESEARCH REPORT -

FISH-BASED REFERENCE MATERIALS

By:

E.G. Bligh and C. Hotton
Canadian Institute of Fisheries Technology
Technical University of Nova Scotia
P.O. Box 1000
Halifax, N.S.
B3J 2X4

Submitted to:

Mr. L. Dalton
Mineral Science Laboratories
Energy, Mines and Resources, Canmet
555 Booth Street
Ottawa, Ontario
K1A 0G1

Under the Terms of:
Canada Supply and Services Contract
21ST 23241-4-1687

December, 1984

MICROMEDIA
MAR 15 1985

6 pp

Preparation of Fish-Based Reference Materials

The objective of this project was to prepare standard reference materials from freshwater fish species for the study of radionuclides in fish.

Approximately 120 kilogram of frozen freshwater fish was provided by the Department of Energy, Mines and Resources for the project. The standard reference materials were prepared according to procedures previously developed at the Canadian Institute of Fisheries Technology for the production of standard reference protein meal and stabilized marine oil from salt water fish species. All materials were handled and processed in accordance with good food manufacturing practice using stainless steel equipment.

Six samples of fish-based reference materials were produced, namely:

1. Protein meal from [illegible] and soft tissue - 9.6 kilogram
2. Bone meal - 4.0 kilogram
3. Skin and scales meal - 4.5 kilogram
4. Oil, extracted from the protein meal - 1.05 kilogram
5. Oil, extracted from the bone meal - 30 gram
6. Oil, extracted from the skin and scales meal - 200 gram

Sample Preparation

Since our equipment did not allow for a good separation of skin from bones during the mechanical separation of the soft tissue, the frozen fish were first completely stripped of their skin and scales by blanching them in live steam for a few minutes.

The stripped fish were allowed to completely thaw at room temperature before being fed through the meat-bone separator equipped with a 5 mm hole drum.

The resulting product containing minced flesh and soft tissue, including that from the internal organs and viscera was packed in plastic bags, re-frozen in 2.25 kilograms waxed cardboard boxes and stored at -30°C for subsequent spray-drying.

Skin and Scales Fraction

The skin and scales recovered from the blanching process were dehydrated in a mechanical air dryer. The dried material was then ground through a 0.030 inch screen followed by two washes with pesticide grade acetone (Fisher Scientific Co., A-40 dimethyl ketone) to remove the fat associated with the skin. The acetone washed powder was vacuum stripped to remove all traces of solvent and finally milled through a 0.024 inch screen to render the powder uniform and free-flowing.

The oil was recovered from the solvent and vacuum stripped to remove all traces of acetone. A simple dilution of the acetone extract with 10% distilled water released the oil which rapidly sank to the bottom of a separatory funnel. An antioxidant, 0.025% of delquin-oil, (100% "ethoxyquin", 2-dihydroxy-6-ethoxy-2,2,4-trimethylquinoline, Delmer Chemicals Ltd.) was added to the oil and mixed thoroughly before bottling.

Bone Fraction

The bones, collected at the waste chute of the meat-bone separator were dried into the mechanical air dryer.

The dried bones were than ground through a 0.030 inch screen followed by two washes with acetone to remove the fat from the small pieces of flesh still attached to the bones and facilitated milling of the powder.

The acetone washed powder was vacuum stripped and milled through a 0.024 inch screen.

A small amount of oil was recovered from the solvent and was treated the same way as the oil from the skin fraction.

Soft Tissue Fraction

The minced flesh was spray dried in four lots of 14.75 kilogram each. Defrosted at +2°C overnight, the minced flesh was homogenized by passing it through a cutting mill equipped with a 0.015 inch cutting head. An equal volume of nearly boiling distilled water was added to the flesh during the homogenization process to coagulate the proteins and prevent gumming and plugging of the liquid distributor during spray drying.

The spray dryer air inlet temperature was set at 190°C and the air outlet maintained at 105-110°C for the duration of the process.

The spray dried powder was extracted three times with pesticide grade acetone to remove fat. The solvent washed powder was vacuum stripped at 30°C to remove all traces of acetone. No further milling was necessary and all four lots of dried powder were mixed together in a stainless steel tumbler for 24 hours at room temperature to yield one large thoroughly mixed batch of protein meal.

The fat was recovered from the solvent by water dilution and after vacuum stripping, 0.025% antioxidant was added to the oil and mixed thoroughly.

Packaging of Samples

The samples of dried powder were sealed in double plastic bags and packed in cardboard boxes for shipment.

The oil samples were poured in glass bottles and each bottle was flushed with nitrogen, securely capped and packed in strong cardboard boxes for shipment.

Care was taken not to contaminate any of the finished samples with foreign materials or dust.

Analytical Data

Samples were collected at various stages of the process for proximate composition analyses of each product (moisture, protein, fat and ash).

Analyses for free fatty acid and peroxide values were also done on two of the oil samples.

Data are summarized in Tables 1 and 2.

TABLE 1

Proximate Composition Analyses (wet basis)

	% Moisture	% Protein	% Ash	% Fat
Raw mince flesh and soft tissue	80.3	14.6	1.7	4.0
Spray dried mince flesh and soft tissue	2.4	59.7	9.2	22.2
Defatted protein meal from flesh and tissue	1.1	71.2	7.5	8.8
Bone meal	2.2	36.0	45.5	9.2
Defatted bone meal	2.0	39.0	49.4	2.5
Skin and scale meal	1.5	59.0	27.8	10.0
Defatted skin + scale meal	1.0	68.0	30.2	1.7

TABLE 2

Oil Analyses

	Free Fatty Acid as % oleic	Peroxide Value m eq/1000 g
Oil from flesh & tissue	0.47±0.014	83.20±1.80
Oil from skin + scale meal	0.785±0.003	19.60±1.81

Process Yield Diagram

The flow diagram of the various steps in the process together with yield figures are shown in Figure 1. The data reflect the actual amount of product obtained after each processing step. The numbers do not always add up because of the loss of material that inevitably occurred through the equipment during the various processes.

Figure 1. Process Flow Diagram and % Yield